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PARTICLE SIZE ANALYZER COMPARISON

L. Kuehn Walker and V.G. Reynolds
Resource Utilization Laboratory

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PARTICLE SIZE ANALYZER COMPARISON

by

L. Kuehn Walker*
and V. G. Reynolds**

SUMMARY

Testwork was done to find a correlation factor between particle-size analyses obtained using five different methods. Size distributions of samples were measured on all particle sizing equipment available at the CANMET Resource Utilization Laboratory, viz.: a Brinkmann Particle Size Analyzer (and Image Analyzer), a Micromeritics Sedigraph® 5000ET, a LEEDS & NORTHRUP Microtrac™, Model 7991-01, and a set of micro screens).

Instruments were compared for reproducibility, using both replicate analyses of the same sample, and fresh aliquots.

The Microtrac™ exhibited the best reproducibility, showing an average standard deviation for repeat tests of ≤1, and <9 for replicate tests. The Brinkmann instrument showed poor repeatability in all respects. No clear correlation between the different methods was discovered. The size distribution obtained, however, was found to depend on the analytical method used, which, in turn, is dependent upon the physical characteristics and composition of the sample.

Keywords: Particle Size Analyzer, Sedigraph, Microtrac, Brinkmann, measurement

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* Physical Scientist, Resource Utilization Laboratory, MSL, CANMET
**Research Officer, Resource Utilization Laboratory, MSL, CANMET

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INTRODUCTION

Over the past two decades, various particle-size analyzers have come on the market. Each analyzer uses a different principle to measure particle size, and each claims to provide the most accurate particle size distribution. In practice, it has been observed that no two instruments give identical size distributions. This has caused confusion and uncertainty for industry when interpreting results from different sources and equipment.

To provide a correlation between the equipment available at CANMET (viz.: a Micromeritics Sedigraph® (Model 5000ET), a LEEDS & NORTHRUP Microtrac™ (Model 7991), a Brinkmann Particle Size Analyzer and Image Analyzer (Model 2010), and a set of micro screens), each apparatus was used to analyze samples of glass beads, sphalerite and hematite, along with a calcite sample which had been classified into "fines", middlings ("mids"), and coarse fractions. Certified standards (three sizes of polystyrene balls) were acquired, but were unsuitable for measurement by the Microtrac™ or Sedigraph® due to the manner in which they were presented.

Each piece of equipment was evaluated for reproducibility of replicate analyses and repeated analyses of fresh aliquots of the same material. Comparisons were made between all the methods.

1.0 DESCRIPTION OF PARTICLE-SIZE ANALYSIS EQUIPMENT

The five methods of particle-size analysis chosen for comparison are those available at the CANMET Resource Utilization Laboratory, where all of these methods are frequently used for ongoing research. Following is a detailed description of each method and a summary of the characteristics of those methods. (Table 1)

1.1 Brinkmann

1.1.1 Particle-Size Analyzer (PSA) (Model #2010)

The Brinkmann Particle-Size Analyzer measures the diameter of a particle directly, using the "measurement of time of transition" theory. This theory states that if a laser scans a field at a fixed velocity, then its time of transit across a particle multiplied by the laser velocity, gives a displacement which is equal to the diameter of the particle[3].

Particle size measurement is performed using a focussed laser beam, collimated to a width of 1 µm, which is scanned circularly at a fixed frequency. Particles are introduced into the measurement area where they interact with the laser beam, producing a voltage pulse in a PIN photodiode mounted behind the measurement area. The width of a pulse represents the time of interaction with a particle and is related to its size, while the rise time of the pulse is shortest for particles in the laser's focal plane, and for those representing a perpendicular interaction with the laser (i.e. the length measured is a diameter). Using proprietary software to select and interpret pulses, this machine is capable of measuring particle sizes between 0.5 μm and 300 μm.

The sample can be introduced to the measurement area in a number of ways. It can be suspended in a transparent liquid and held in a transparent, 4-mL cuvette containing a small magnetic stirrer, or a suspension can be treated using a "flow-through" (LQF) cell and variable-speed peristaltic pump. For particles with high specific gravities, a viscous fluid such as glycerine or glycol may be used to prevent rapid settling. The cuvette and its associated magnetic stirrer cannot be used, of course, when magnetic particles are present, as they would adhere to the stirrer.

The sample can also be mounted on a microscope slide. Then, during analysis, a slide-holder assembly allows vertical movement of the slide through a distance of 1 cm, presenting several "fields" per slide.

1.1.2 Image Analyzer

The Brinkmann Image Analyzer performs basic shape analysis of an image using an IBM XT computer with 640 kilobytes of random-access memory (640kb RAM). A sample is introduced in the same manner as for particle sizing, and an image of the sample is acquired using a CCD (charge-coupled device) camera. This 256-grey-level image is then displayed on a monitor having a resolution of 500 x 400 pixels. By the use of subroutines and menu functions, certain objects can be smoothed, "segmented" and/or selected by manipulating grey levels, while other (spurious) objects are rejected.

The image can then be converted to one consisting of black objects on a white field by specifying a cutoff grey level above which every pixel is converted to white, and below which every pixel is converted to black. At this point each black object present can be analyzed for many different properties such as length, width, shape factor, area, etc. Individual measurements for each object (feature specific), and <u>global</u> measurements for <u>all</u> objects (field specific) in the image can be made[8]. In addition, a <u>series</u> of images can be analyzed and the data combined, to increase the size of the sample set.

1.2 Micromeritics Sedigraph® (5000ET)

The Micromeritics Sedigraph® employs a sedimentation technique whereby the measured equilibrium velocity of a particle falling through a viscous medium, due to gravitational force, can be related to the <u>size</u> of the particle by Stokes' Law[4].

$$D^2 = \frac{18v\eta}{(\rho-\rho_0)g}$$

D = particle diameter
v = particle velocity
η = liquid viscosity
ρ_0 = liquid density
ρ = particle density

The sedimentation velocity can be determined by measuring the concentration of particles remaining in suspension as a function of time. Stokes' Law, however, is based on spherical particles. Thus, when dealing with irregularly-shaped particles, the measured diameter is the diameter of a sphere of the same material having the same sedimentation velocity. This definition is known as the "Stokes diameter".

A sample, dispersed in a suitable suspending medium, is circulated through a measurement cell using a peristaltic pump. At the analysis start time, circulation is stopped, allowing the particles

to settle. The cell is irradiated perpendicularly by a finely-collimated X-ray beam. The degree of attenuation of incident radiation by the suspension, relative to the suspending fluid alone, is a function of the weight concentration of suspended solids.

The method requires only a few grams of sample, and plain water is often a suitable suspension medium. In addition, the method is capable of measuring particles from 0.13 μm to 100 μm, with very good resolution in the finer ranges.

On the other hand, the higher the density of the sample, the smaller the maximum particle size that can be measured (in a given medium). Moreover, to suspend particles of high specific gravity for a suitable measurement time requires the use of a dense suspension fluid whose density and viscosity at the ambient temperature are known. Furthermore, the specific gravity of the sample must always be known, - a severe limitation for mineral processing purposes, since only homogeneous samples may be analyzed. If a denser suspension medium is required, operation tends to be more cumbersome.

1.3 LEEDS & NORTHRUP Microtrac™ (Model 7991)

Utilizing a light-scattering technique, the Microtrac™ laboratory particle-size analyzer is capable of analyzing particles within the range of 2 to 176 μm. Its operation is based on the Fraunhofer diffraction theory, which states that the intensity of light scattered by a particle is directly proportional to the particle size; large particles scatter light at high intensities through low angles, while small particles scatter light at low intensities through high angles.

Particles circulating through a cell are illuminated by a helium-neon laser beam (λ = 0.6328 μm). The resulting Fraunhofer diffraction pattern is collected (using a series of lenses and a proprietary "Spatial Filter") by a photodetetor connected to a microprocessor which processes the data[1,2]. The particle diameters given by this method are the "equivalent spherical diameters".

The instrument has a lower detection limit of 2 microns due to its inability to detect low-intensity scattered light from particles this size and smaller. This is an important limitation because the size distribution is calculated on a "100%-of-particles-detected" basis. If particles smaller than 2 microns are present, the size distribution will be biased towards the coarse sizes. To overcome this limitation, a method has been developed by W.H. Cameron et al.[2] to determine the fraction of sample material which is less than 2 microns in size. This value can then be used to adjust the measured size distribution.

1.4 Micro Screens

Particle-size analysis using micro screens is identical to conventional wet screening except that alcohol (rather than water) is used as the dispersant, to prevent agglomeration of the fine particles. When a slurry is introduced to a screen having a fixed aperture size, particles which are finer than the screen opening will pass through, leaving the coarser particles on the screen.

Surface tension effects slow the passage of particles through the screens. Also, a layer of larger particles soon develops next to the screen surface, and this further slows the passing of finer particles, making the method very time consuming.

Good results require good operator technique. It is especially important to check each screen periodically for small tears which, if unnoticed, would skew the results toward the finer end of the scale. Also, except for spherical particles, results are time-dependent. Micro screening measures a particle's smallest continuous diameter, since only one dimension as small as the screen opening is required for the particle to pass through the screen. Thus with agitation and time, more and more oblong particles whose lengths are close to but greater than the aperture size, and whose long axes are at first parallel to the screen surface, become so oriented that they may pass through the screen.

Table 1: Characteristics of CANMET Particle Sizing Equipment

	Sedigraph®	Microtrac®	Brinkmann	Image Analysis	Screens
Measurement Method:	Sedimentation	Light Scattering	Time of Transit	Projected Image	Physical
Parameter Measured:	Stokesian Diameter	Equivalent Spherical Diameter	Projected Equivalent Dia.	Average Ferets Diameter	Smallest Diameter
Size Range (μm):	0.13-100	2.0-176	0.5-300	2-1200	2 - >1200
Sample Type:	Homogeneous	Heterogeneous	Heterogeneous	Heterogeneous	Hetero-geneous
Mass of Sample:	~10g	>3g	Cuv.*: <0.5g LQF**: ~3g	<0.5g ~3g	<3g

*transparent cuvette
**Liquid flow cell

2.0 DESCRIPTION OF SAMPLES

The test samples were chosen to reflect the range of materials which a particle-size analyzer might be called upon to measure in a mineral processing laboratory setting. For simplicity of comparison and analysis, homogeneous samples were used.(Note the requirement of the Sedigraph® for homogeneous samples, p.4.) A comparison of sample characteristics appears in Table 2.

2.1 Calcite

A suitable, homogeneous calcite sample was selected, ground dry, and then air classified into "heads", "coarse", "mids", and "fines" fractions, in the CANMET Industrial Minerals Section. The specific gravity of calcite is 2.7.

2.2 Potter Industries Glass Beads (#3000)

These beads are spherical in shape, with a maximum diameter of 44 µm, as measured by a Coulter Counter®. Their usual application is in the testing of aircraft specifications; - hence no size range is specified. Their sphericity eliminates the size measurement problems associated with the shape of particles, yet since they are glass, they are transparent or translucent to some analyzers. The specific gravity of glass is approximately 2.6.

2.3 Sphalerite

A 554.5g sample of pure sphalerite was ground for 30 minutes, at 65% solids, in a ceramic ball mill containing a 5-kg ceramic ball charge. After grinding, 96% of the product passed a 150M (104 µm) screen. This sample was chosen for its intermediate specific gravity of 4.1 and its non-spherical particle shape.

2.4 Hematite

The same procedure used to produce the sphalerite sample was employed for this sample, except that only 502g were ground. The product was found to be 97% passing 150M (104 µm). Hematite has a specific gravity of 5.2.

2.5 Polystyrene Standards

Eight sizes of spherical, polystyrene standards were purchased from Coulter Electronics of Canada (who also manufacture particle sizing equipment). These standards, consisting of single-size beads suspended in a water-miscible dispersant, are packaged in 15-mL vials. Three sizes were used in these tests, viz.: 21.36 µm, 45.61 µm and 86.81 µm. The specific gravity of polystyrene is approximately 1.0.

Table 2: Comparison of Sample Characteristics

	Specific Gravity	Aspect Ratio	Shape Factor
Calcite Fines	2.7	0.69	0.74
Calcite Mids	2.7	0.72	0.78
Calcite Coarse	2.7	0.69	0.76
Calcite Heads	2.7	0.68	0.74
Glass Beads	2.6	0.89	0.91
Sphalerite	4.1	0.69	0.75
Hematite	5.2	0.69	0.76
Standard 21.36 μm	1.0	0.77	0.80
Standard 45.61 μm	1.0	0.87	0.88
Standard 86.81 μm	1.0	0.88	0.83

NOTE: Aspect ratios and shape factors were measured using the Brinkmann Image Analyzer. (See Appendix D for definitions of terms.)

3.0 SAMPLE PREPARATION

All samples, with the exception of the polystyrene standards, were prepared in the same manner, regardless of the analysis method to be used. Approximately 5g of material was added to 20 mL of distilled water. The tip of a high-energy ultrasonic probe was then submersed in the slurry, and the probe was allowed to operate for 3 minutes in order to eliminate the agglomeration of particles.

For the Sedigraph®, Microtrac™, and screens, the entire mixed sample was used for the analysis. For the Brinkmann instrument, however, only a small portion of the prepared sample was required for cuvette analysis. While the bulk sample was vigorously agitated using a magnetic or mechanical stirrer, the needed amount was withdrawn using a small pipette. When the "liquid flow" (LQF) cell was used, the circulating sample was agitated in the "sump" with either a magnetic or a mechanical stirrer.

4.0 SAMPLE ANALYSIS

Analyses involving the Brinkmann LQF cell utilized a peristaltic sample-recirculating pump. Dial settings of 3.5, 7, and 10 were used in these tests, and correspond to pump outputs of 130, 330, and 500 mL per minute, respectively.

4.1 Calcite

All calcite samples were analyzed 3 times on the Microtrac™ instrument, and the results were averaged. Analyses were performed only once on all other equipment. The Brinkmann analyses were performed with both the cuvette cell, using a stirrer speed of 3 (i.e. maximum), and the LQF cell (where the peristaltic pump speed was set at an intermediate, unrecorded, value). A reading for the coarse sample was unobtainable on the Sedigraph® due to its coarse size. Results varied quite substantially, as can be seen from the summary tables (## 8,9,10,and 11 in Appendix B).

4.2 Glass Beads

The analysis of glass beads was successful with all equipment. It was hoped that this sample would be light enough to remain in suspension while being analyzed in the cuvette cell of the Brinkmann Particle-Size Analyzer, but it was found that settling did occur. For that reason, all further analyses were done using the LQF cell. A summary of the analytical results appears in Table 12, Appendix B.

4.3 Sphalerite

The analysis of sphalerite on all the equipment was very straightforward. To accommodate the high specific gravity of sphalerite, however, the Sedigraph® required the use of

Sedisperse® A-13, a dispersion liquid supplied by Micromeritics, having a viscosity approximately ten times that of water.

During the Brinkmann analyses, some settling or adhesion to the walls of the Tygon® tubing was encountered, which could not be eliminated by increasing the pump speed. A summary of the analytical results appears in Table 13, Appendix B.

4.4 Hematite

As mentioned above, some problems with settling or adhesion of particles to tubing walls were experienced with the Brinkmann LQF cell. Also, since hematite is magnetic, a mechanical stirrer was used for all analyses done with the Brinkmann PSA. It is not possible, however, to operate the Sedigraph® without using a magnetic stirrer. As a result, some of the hematite was attracted by the magnetic stirring bar and was not analyzed, rendering analyses invalid. The other methods produced satisfactory results.

4.5 Polystyrene Standards

The polystyrene standards were analyzed using the Brinkmann PSA and Image Analyzer only, since there was insufficient sample available to produce the required slurry density for the Sedigraph®, and the dispersant in which the standards were packaged caused entrainment of air bubbles in the sample stream of the Microtrac™. Replicate analyses of the same aliquot were carried out using the Brinkmann cuvette sample cell (to conserve sample), with stirrer speeds of 2 and 3. The results obtained are shown below.

Table 3: Analysis of Standards Using the Brinkmann PSA

Run #	Stirrer Speed	21.36 µm Mean	21.36 µm Std.Dev.	45.61 µm Mean	45.61 µm Std.Dev.	86.81 µm Mean	86.81 µm Std.Dev.
1	2	22.66	7.88	47.33	16.45	82.25	19.83
2	3	21.75	6.62	45.32	16.88	70.13	23.51
3	3	22.42	8.56	42.36	13.61	57.40	23.78
Image	3	22.00	---	45.40	---	83.00	---

For the smallest spheres, the stirrer speed appeared to have no bearing on the analysis; for all speeds and methods the results were within 6% of the nominal value.

For the 45.61 µm standard, the results were within 7% of the nominal size, while image analysis showed a very small difference of only 0.4%.

Difficulties were encountered in maintaining the 86.81 μm particles in suspension. In addition, successive runs showed progressively smaller sizes. Image analysis revealed fractured particles, apparently caused by vigorous agitation of the sample. The same effect was seen in the case of the 45.61 μm particles, but to a lesser extent. Although a fresh sample was taken for image analysis, the mean particle size measured was lower than the nominal, - a further indication of particle deterioration due to agitation during the analysis.

Both the image analyzer and the particle size analyzer produced acceptable readings deviating no more than 7% from the nominal size. The operator should be aware, however, that as the particle size becomes larger, greater care is required to ensure that the particles are kept in suspension while the concomitant attrition is maintained at an acceptably low level.

5.0 REPRODUCIBILITY OF RESULTS

5.1 Replicate Analyses - (Same Sample)

The observed variation of standard deviation as a function of particle size, in replicate analyses of the same sample, is shown in Figures 1, 2, and 3. It can be seen that the magnitude of the standard deviation is largely dependent upon the type of material analyzed.

The Microtrac™ is the most consistent instrument, with a standard deviation of ≤1 for all samples and particle sizes. Its consistency is likely due to its method of operation; i.e. it processes the largest amount of sample and has the best agitation system. Thus, a more representative sample is obtained, lowering the variation between measurements.

The Sedigraph® exhibited a bell-shaped curve for all samples, with maxima of 2.5, and 1.5 for glass beads and sphalerite, respectively. Bell-shaped curves have been observed by others for other X-ray sedimentometer analytical methods, but no conclusions have been drawn relating the shape and maximum of a curve to particle size[6].

The Brinkmann PSA was much less consistent, with higher standard deviations seen in the mid-size ranges. Standard deviations produced by the image analyzer increased with particle size. This may be due to the difficulty in focussing clearly on large particles. For example, if a large, oblong particle is oriented obliquely to the axis of the camera, some portion of the particle will always be out of focus and thus excluded from the image. This phenomenon was most pronounced in the case of hematite where particle shape often appeared irregular during analysis.

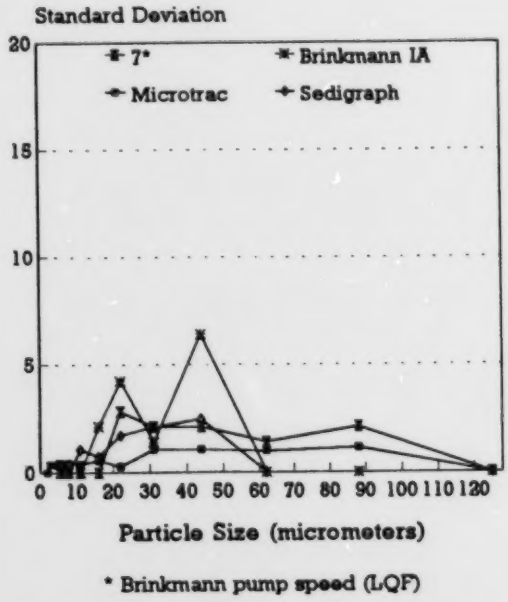
5.2 Repeat Analyses - (Fresh Samples)

Because a representative sample is essential for good particle size analysis, those techniques requiring relatively large samples, (i.e. the Brinkmann LQF cell, the Microtrac™, and the Sedigraph®) are most likely to give analyses with low standard deviations, while the Brinkmann instrument using the cuvette and image analysis methods calls for meticulous dilution technique and multiple runs in order to obtain comparable statistics. The Brinkmann instrument, however, even using the LQF cell, fared poorly in comparison with the other methods, with standard deviation maxima of 30, 11, and 42 for glass beads, sphalerite, and hematite, respectively (Figures 4, 5, and 6).

Due to their sphericity, the glass beads were analyzed without difficulty - with standard deviations less than 4 - by both the Microtrac™ and Sedigraph®, while their sphalerite and hematite analyses showed standard deviations of 9 or less.

Figure 1:

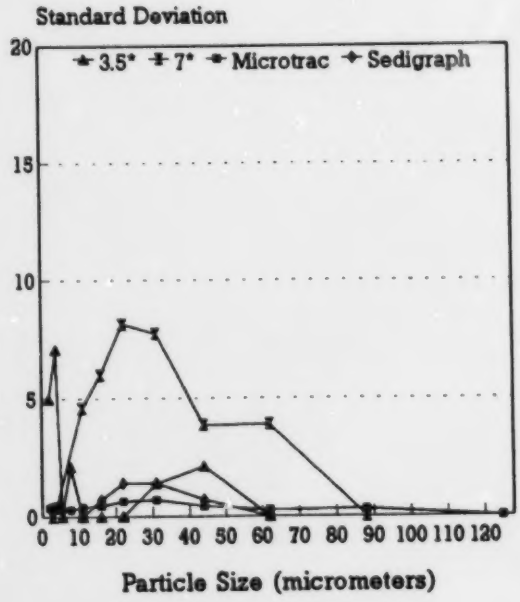
Standard Deviation
Glass Beads - Same Sample



* Brinkmann pump speed (LQF)

Figure 2:

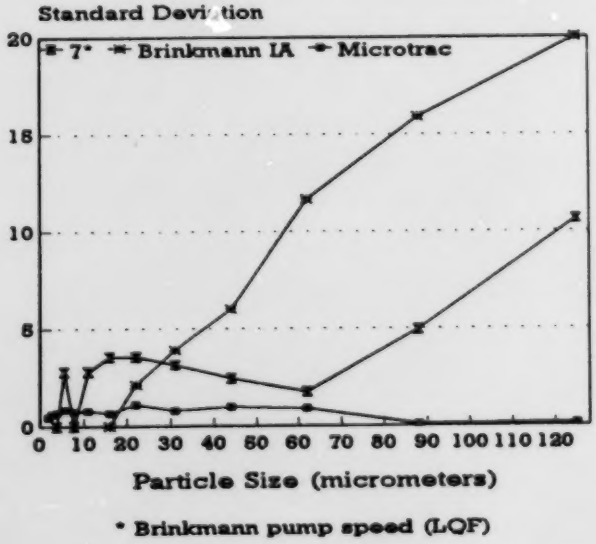
Standard Deviation
Sphalerite - Same Sample



* Brinkmann pump speed (LQF)

Figure 3:

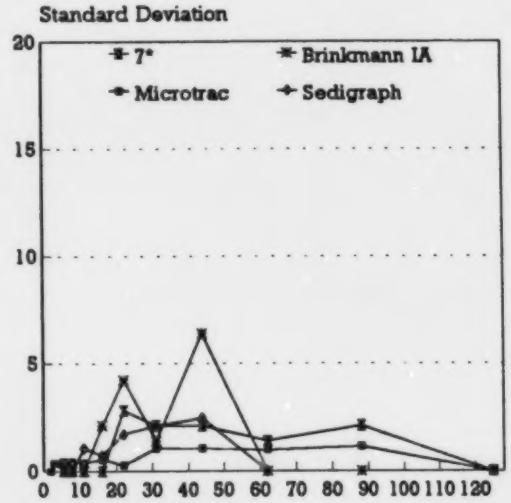
Standard Deviation
Hematite - Same Sample



* Brinkmann pump speed (LQF)

Figure 1:

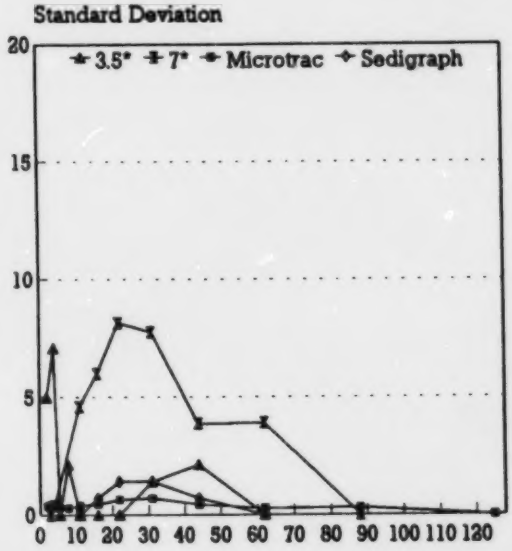
Standard Deviation
Glass Beads - Same Sample



* Brinkmann pump speed (LQF)

Figure 2:

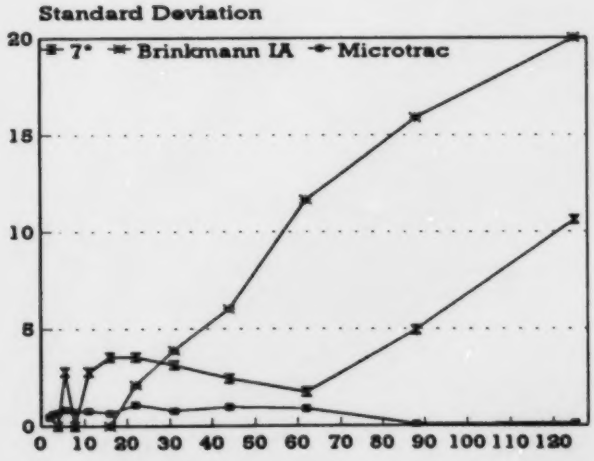
Standard Deviation
Sphalerite - Same Sample



* Brinkmann pump speed (LQF)

Figure 3:

Standard Deviation
Hematite - Same Sample



* Brinkmann pump speed (LQF)

6.0 CORRELATION BETWEEN MACHINES

The results of these tests show that there is no clear correlation between the analytical methods tested, but rather the results vary irregularly, depending upon the sample material.

On log-normal graphs, parallel curves are indicative of instruments which measure identical size distributions, while lateral shifts of those curves (along the x-axis) show discrepancies in measurements of the size of particles in a given size fraction. Such discrepancies could be easily resolved by applying a suitable correction factor. Differences in the shapes and/or slopes of curves, however, indicate fundamental differences which are not easily reconciled.

Predictably, the best agreement was found when measuring uniform spheres such as glass beads. Figure 11, for example, shows good agreement between instruments (slopes and shapes of curves are almost identical). Although the results for the calcite mids and coarse samples were excellent (Figures 8 and 9), the fines revealed some lateral shifting and disagreement in the sizing of larger particles (Figure 7).

The calcite head, sphalerite, and hematite samples were unclassified material, and the wider size range is reflected in shallower slopes and poorer agreement between methods (Figures 10, 12, and 13). The irregularity of shapes and the different surface characteristics of these particles also contributed to this variation in the analytical curves.

It is apparent from an examination of Figures 7 through 13 that the Brinkmann image analyzer differs from the other instruments in two ways. First, it represents a given distribution as being coarser than the other instruments (curves are shifted to the right), and second, it shows a given size distribution as being narrower than the other methods. For example, when the graphical slopes are measured between the d_{50} and d_{75} (Table 4), the Brinkmann I.A. curve is steeper in every case except for the glass beads.

The Microtrac™, Sedigraph® and screens generally gave comparable results. In some cases, the image analyzer and micro screens were also very similar, but the screens always measured finer particles.

The speed of the circulating pump in the Brinkmann PSA had a marked effect upon the distribution curves. For glass beads, as the speed increased, the curve shifted toward the finer sizes. This is clearly not a suspension effect but is probably related to the way in which the software interprets transparent particles. In an earlier version of this software, in fact, such particles were counted as two smaller particles: - one when the traversing laser beam encountered the refractive leading edge of the particle, and the other at the trailing edge. At higher particle speeds the software may lose its ability to respond appropriately to transparent particles. The convergence of the three (3.5, 7, and 10) "speed" curves around the 4 to 5μm region would support this theory.

The sphalerite and hematite samples, being opaque, are not affected in this way. Instead, their much higher specific gravities tend to cause difficulties in maintaining the particles in suspension. In both cases, increasing the pump speed from 3.5 to 7 causes more large particles to remain in suspension, and a corresponding shift in the response curve toward larger particle sizes. When the speed is increased further to 10, the sphalerite size distribution curve appears to shift again to the finer sizes (although the effect is almost negligible for hematite). This is obviously not due to particles dropping out of suspension, but again seems to be related to the speed at which particles pass the detector (cf. comments regarding glass beads, p.14). It appears that the software cannot cope with the fast-moving particles, and under these conditions the message **"SAMPLE TOO CONCENTRATED OR SPECIAL"** is displayed for the operator.

Both the cuvette and the flow-through (LQF) cells were used in the Brinkmann instrument for calcite analysis. The measurements for calcite fines were very similar, but for coarser fractions, the cuvette size distribution curve was shifted toward the finer sizes, indicating insufficient agitation. This occurred in spite of the low specific gravity of calcite and the use of the maximum agitator speed setting.

Figure 7:

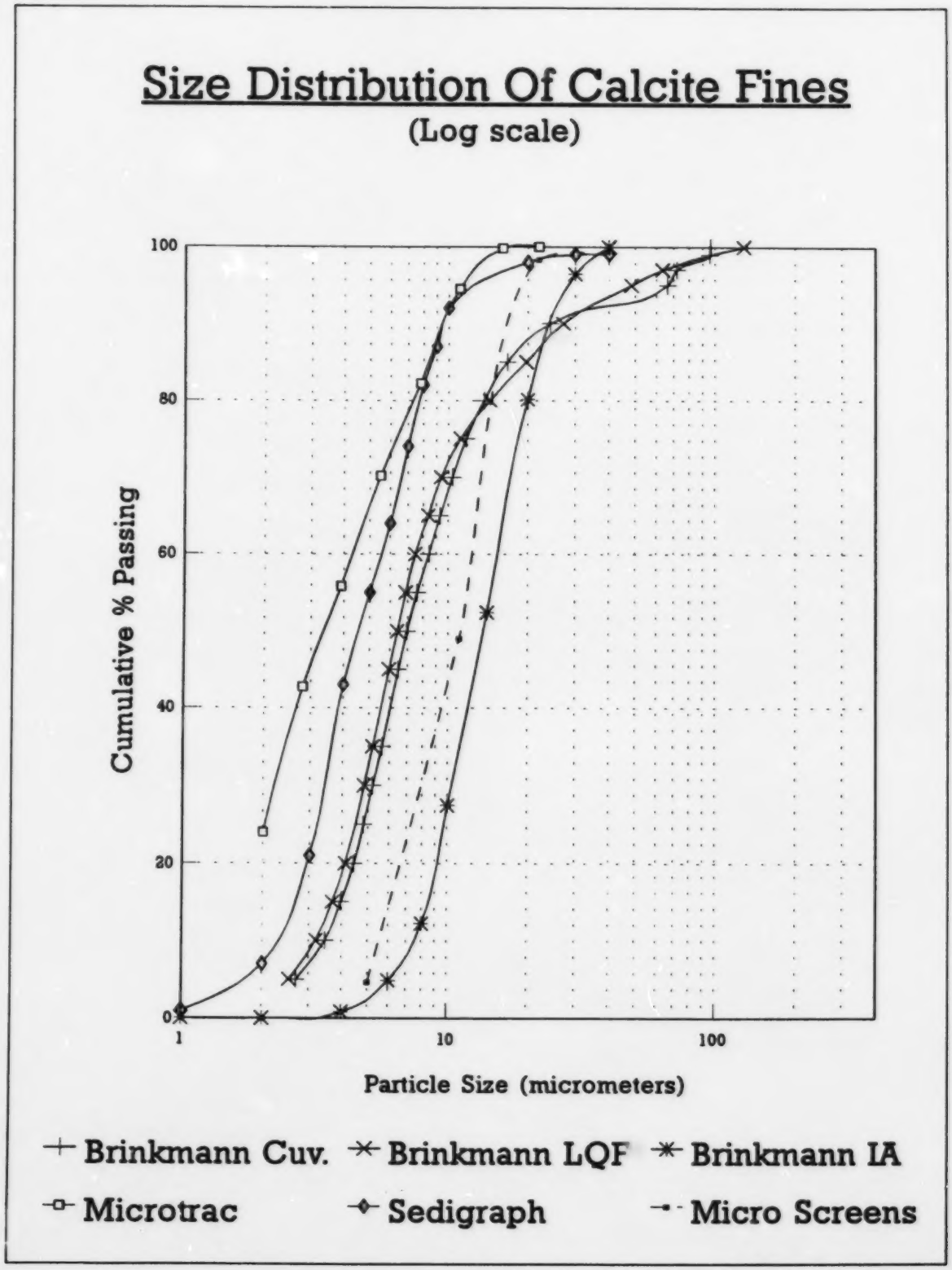

Size Distribution Of Calcite Fines
(Log scale)
Cumulative % Passing
100
80
60
40
20
0
1
10
100
Particle Size (micrometers)
Brinkmann Cuv.
Brinkmann LQF
Brinkmann IA
Microtrac
Sedigraph
Micro Screens

Figure 8:

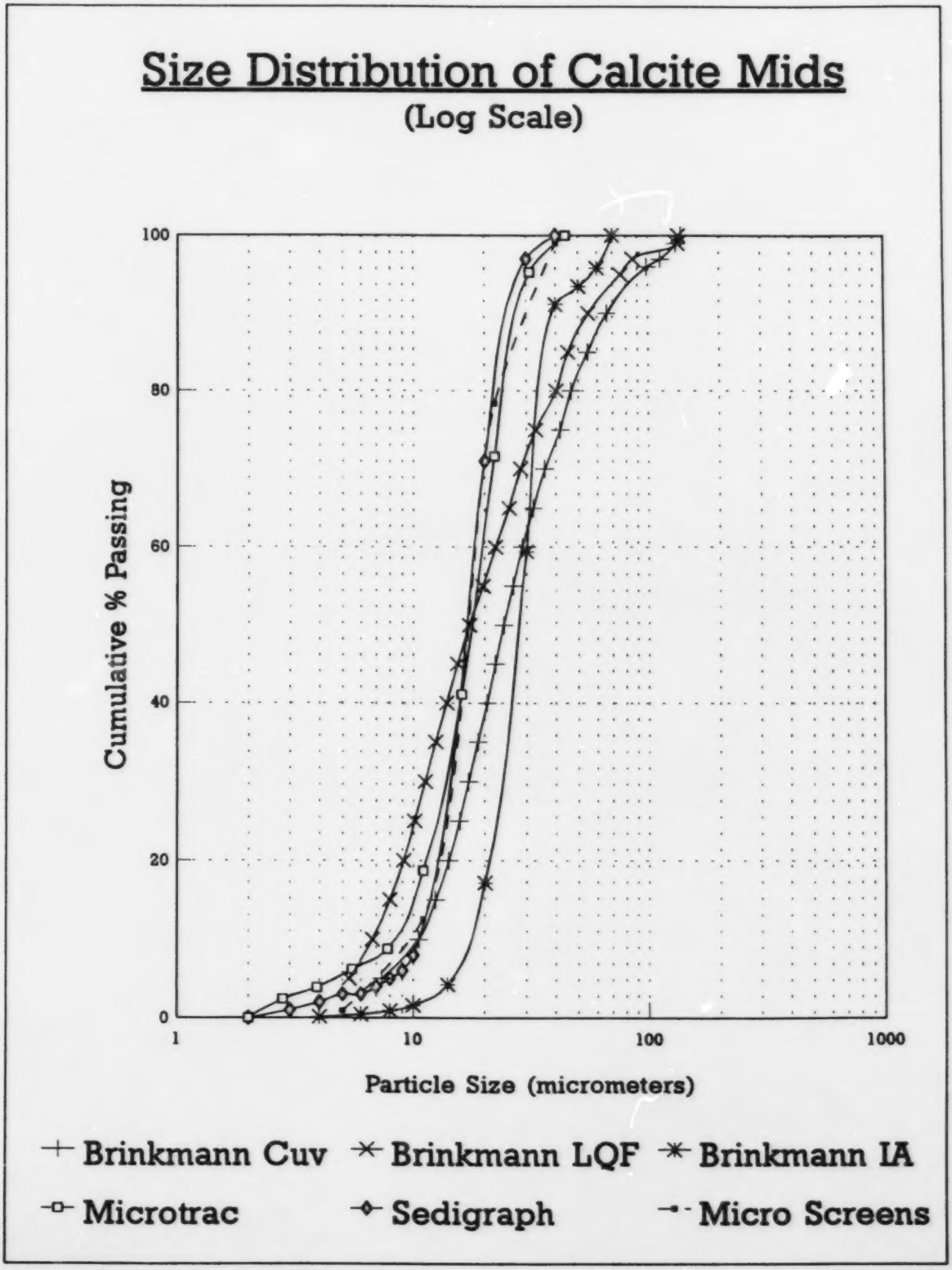

Size Distribution of Calcite Mids
(Log Scale)
Cumulative % Passing
100
80
60
40
20
0
1
10
100
1000
Particle Size (micrometers)
Brinkmann Cuv
Brinkmann LQF
Brinkmann IA
Microtrac
Sedigraph
Micro Screens

Figure 9:

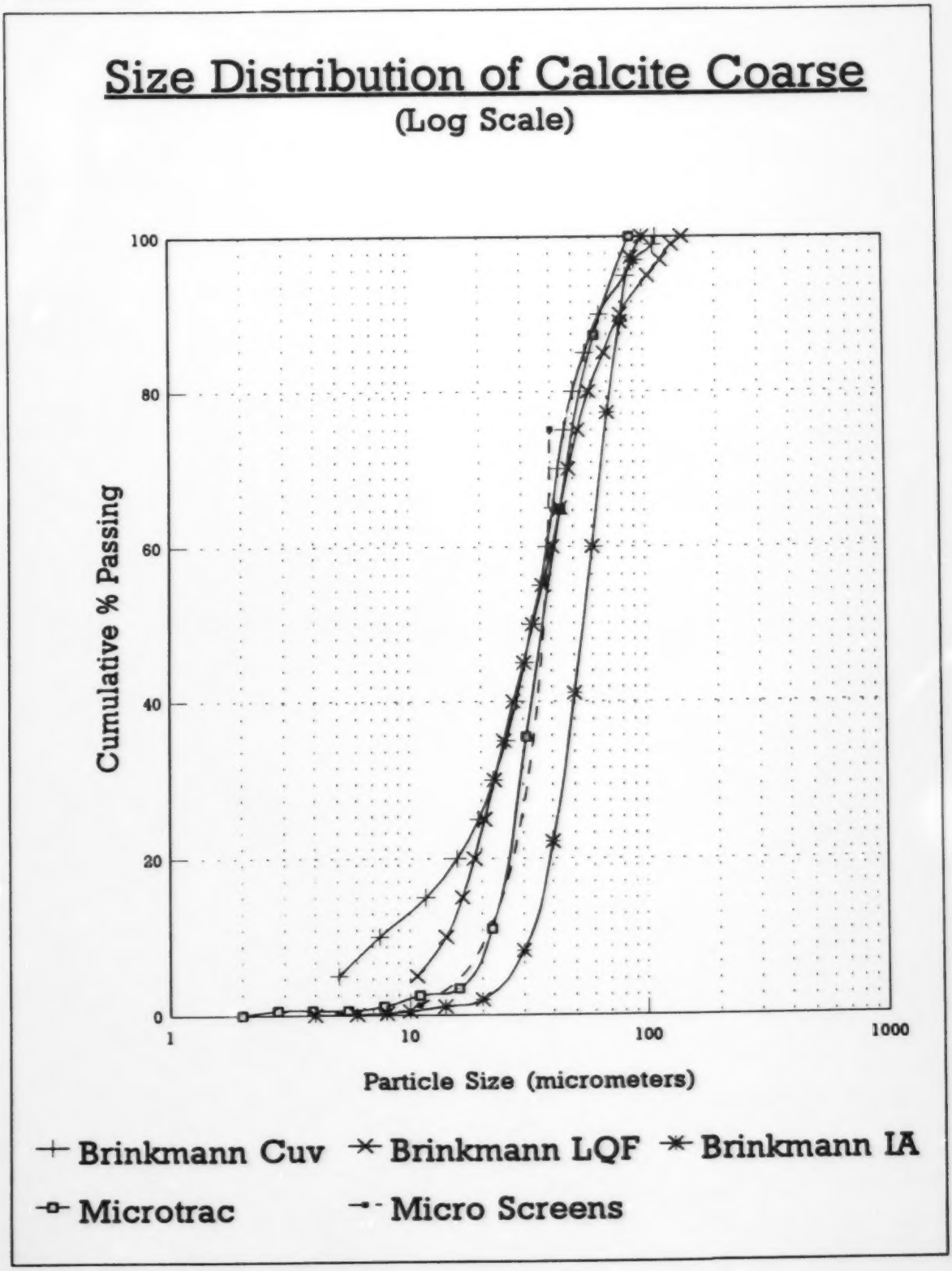

Size Distribution of Calcite Coarse
(Log Scale)
Cumulative % Passing
100
80
60
40
20
0
1
10
100
1000
Particle Size (micrometers)
Brinkmann Cuv
Brinkmann LQF
Brinkmann IA
Microtrac
Micro Screens

Figure 10:

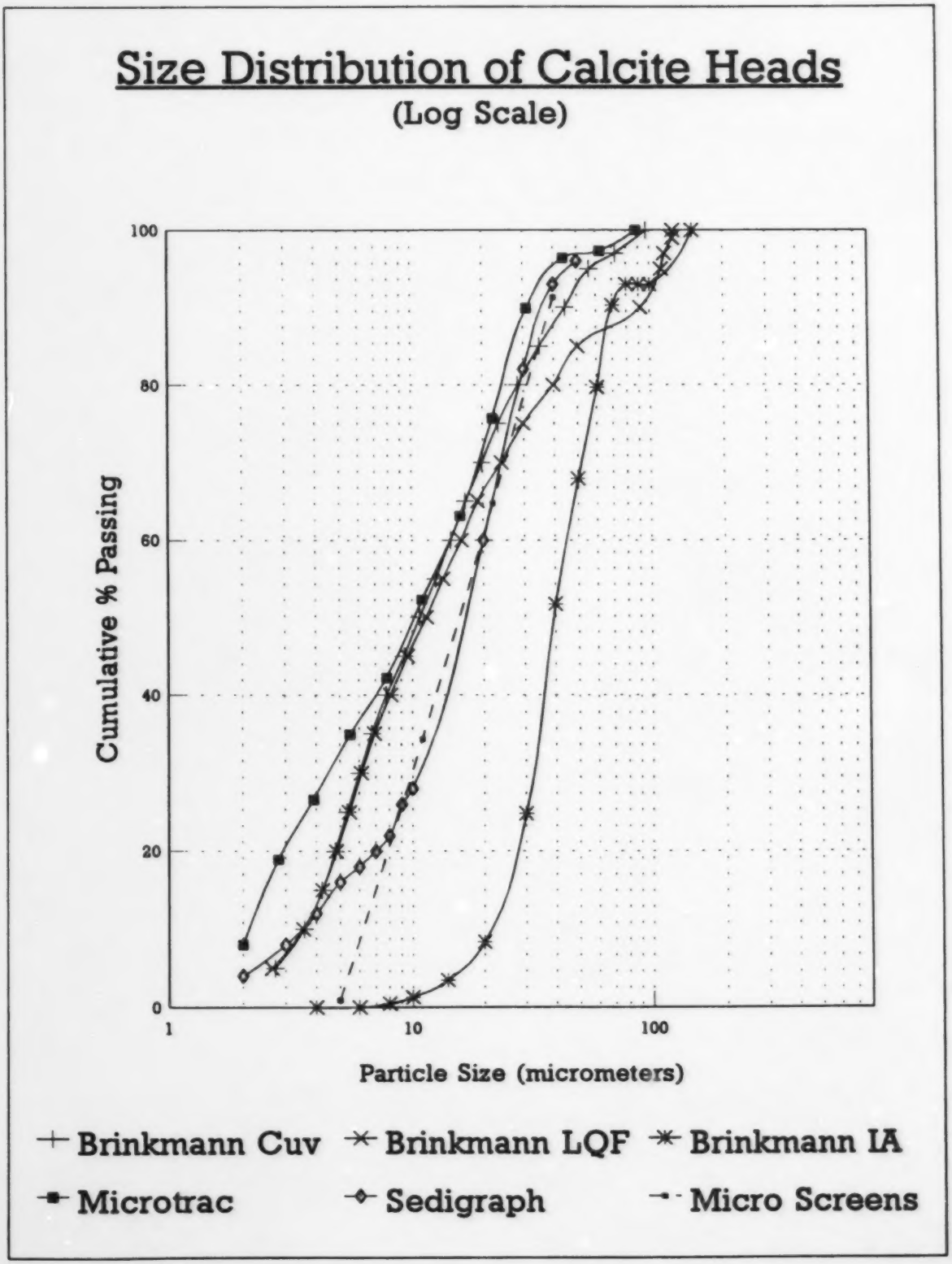

Size Distribution of Calcite Heads
(Log Scale)
Cumulative % Passing
100
80
60
40
20
0
1
10
100
Particle Size (micrometers)
Brinkmann Cuv
Brinkmann LQF
Brinkmann IA
Microtrac
Sedigraph
Micro Screens

Figure 11:

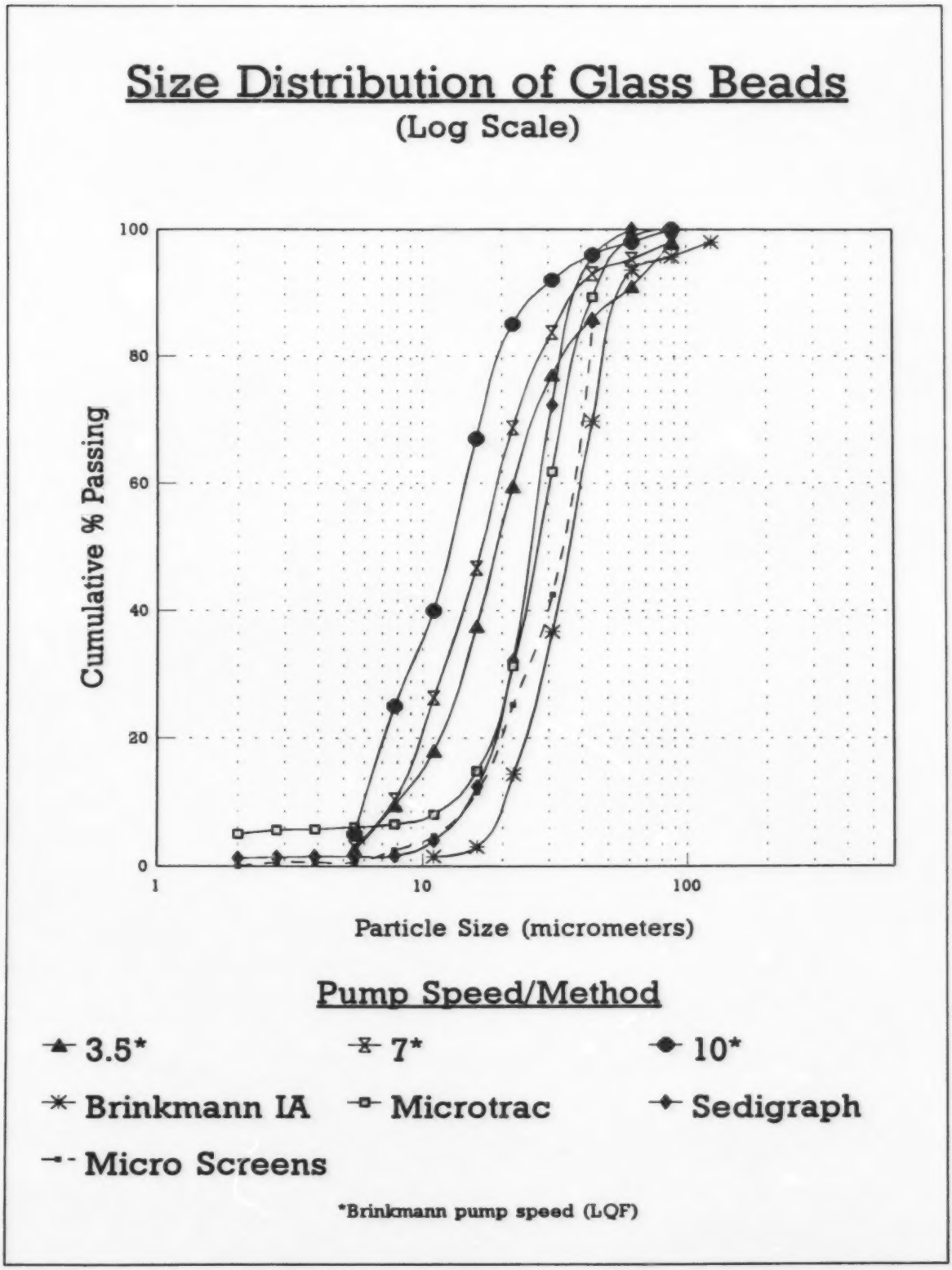

Size Distribution of Glass Beads
(Log Scale)
Cumulative % Passing
100
80
60
40
20
0
1
10
100
Particle Size (micrometers)
Pump Speed/Method
3.5*
7*
10*
Brinkmann IA
Microtrac
Sedigraph
Micro Screens
*Brinkmann pump speed (LQF)

Figure 12:

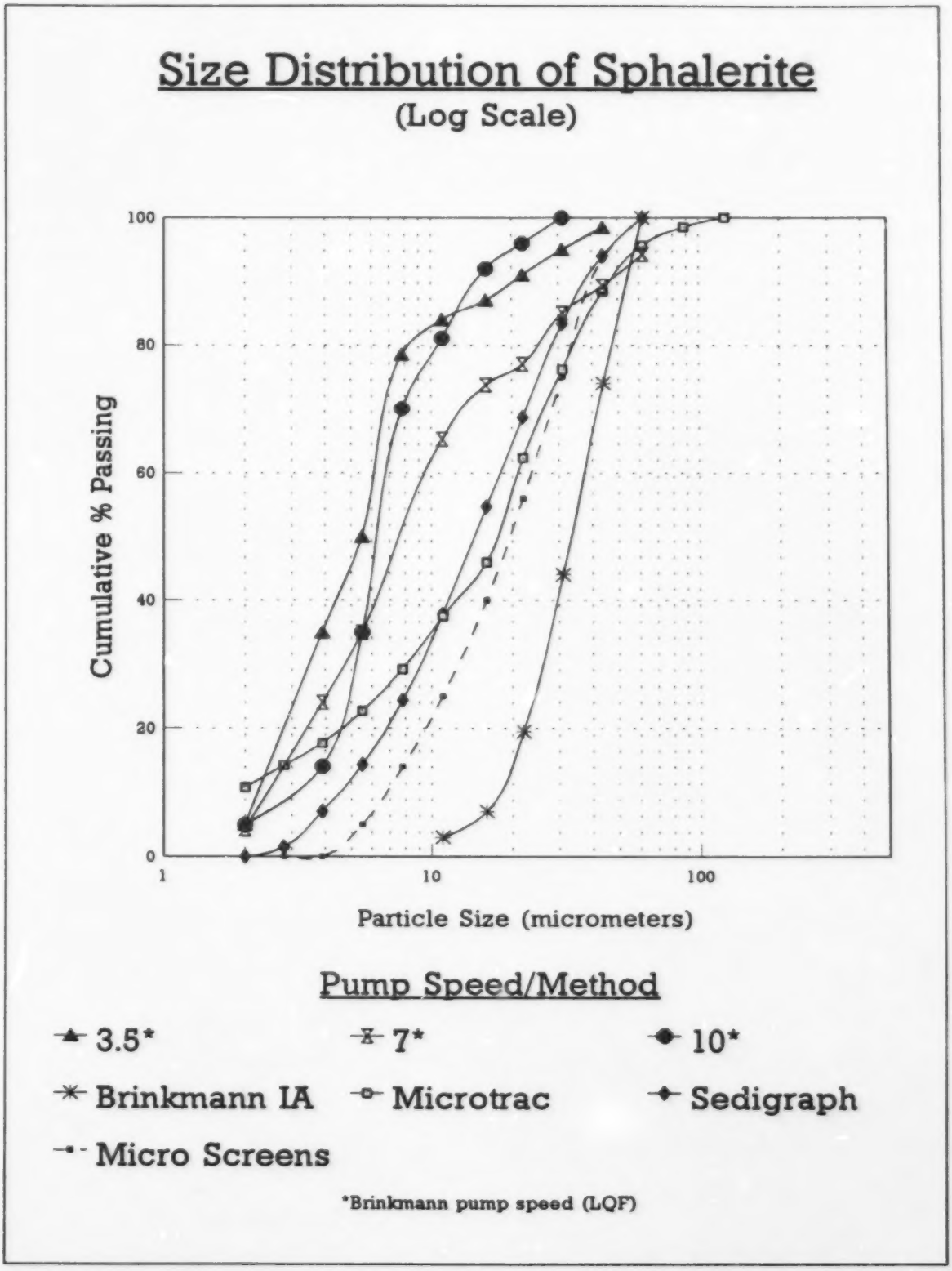

Size Distribution of Sphalerite
(Log Scale)
Cumulative % Passing
100
80
60
40
20
0
1
10
100
Particle Size (micrometers)
Pump Speed/Method
3.5*
7*
10*
Brinkmann IA
Microtrac
Sedigraph
Micro Screens
*Brinkmann pump speed (LQF)

Figure 13:

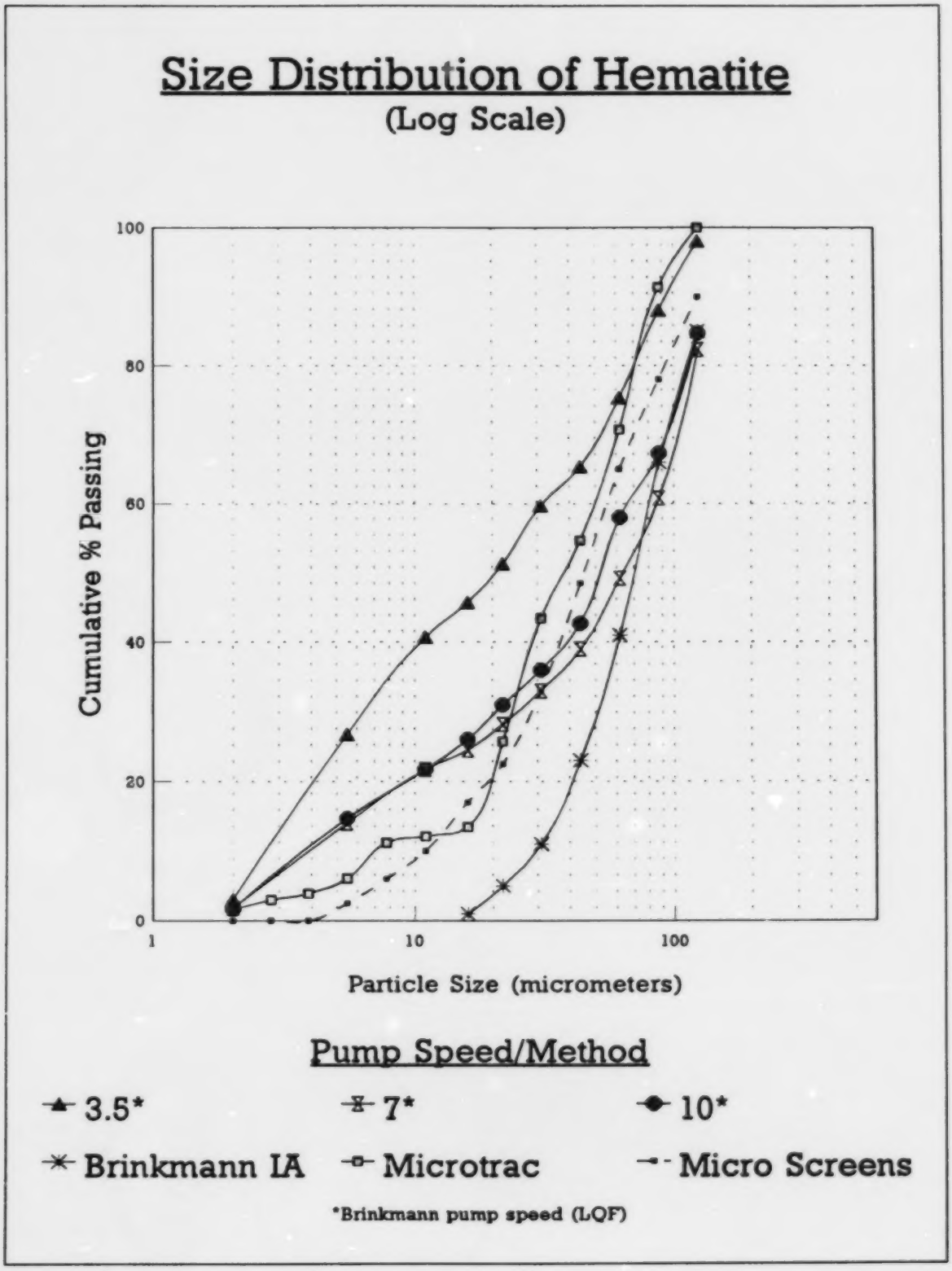

Size Distribution of Hematite
(Log Scale)
Cumulative % Passing
100
80
60
40
20
0
1
10
100
Particle Size (micrometers)
Pump Speed/Method
3.5*
7*
10*
Brinkmann IA
Microtrac
Micro Screens
*Brinkmann pump speed (LQF)

Table 4: Graphical Slopes for all Analyses

	Brinkmann 3.5* (LQF or Cuv)	Brinkmann 7* (LQF)	Brinkmann 10* (LQF)	Brinkmann Image Analyzer	Sedigraph	Microtrac	Micro Screens
Calcite Heads	1.87	1.53		3.62	2.90	1.93	2.64
Calcite Coarse	4.46	3.22		5.80		4.14	5.80
Calcite Mids	2.59	2.28		6.33	4.38	4.75	3.56
Calcite Fines	2.76	2.52		3.87	3.22	2.32	3.41
Sphalerite	3.25	1.53	3.47	4.30	2.26	3.05	2.60
Hematite	1.13	2.08	1.85	3.33	2.27	2.27	2.17
Glass Beads	2.72	2.88	3.27	4.08	4.90	4.08	4.90

* Pump speed

Table 5: Mathematical Slopes for all Analyses

	Brinkmann 3.5* (LQF or Cuv)	Brinkmann 7* (LQF)	Brinkmann 10* (LQF)	Brinkmann Image Analyzer	Sedigraph	Microtrac	Micro Screens
Calcite Heads	73.8	68.8		171.1	191.8	73.0	171.1
Calcite Coarse	119.4	129.7		238.7		85.3	257.0
Calcite Mids	116.4	94.9		257.9	180.0	200.1	141.9
Calcite Fines	92.9	100.8		161.4	137.1	92.9	147.3
Sphalerite	144.2	69.9	156.5	85.8	104.7	101.3	126.0
Hematite	53.2	97.6	83.0	161.4	107.8	107.2	100.0
Glass Beads	86.4	135.5	133.6	221.2	241.5	182.7	299.0

* Pump speed

7.0 CONCLUSIONS

1. The Microtrac™ gave the best reproducibility when analyzing either a fresh sample of a given material, or the same sample repeatedly. (Figures 1 - 6)

2. The Sedigraph® produced its highest standard deviations in the 30μm region, whether replicates were made using the same sample or fresh material. (Figures 1 - 6)

3. The Brinkmann particle size analyzer showed very poor reproducibility for both repeat analyses of samples and for fresh samples. (Figures 1 - 6)

4. The standard deviation for the image analyzer generally increased with particle size.

5. Image Analysis consistently showed the coarsest size distributions (i.e. the distribution curve was shifted toward coarser sizes). This instrument also had the highest resolution (i.e. the steepest slopes on the cumulative size distribution curves).

6. The speed at which a sample is agitated and circulated through the Brinkmann particle size analyzer is very important, and should always be optimized, - especially before lengthy analyses.

7. The physical characteristics of measured particles, such as shape, crystal structure, and surface smoothness, profoundly affect the size distribution measured by the methods investigated in this report.

8. The Brinkmann Particle Size Analyzer and Image Analyzer results agree well with nominal (manufacturer's) values when analyzing ideal (spherical, light, and opaque) samples such as polystyrene balls. (Table 3)

9. Other laboratories have also found good instrumental agreement for spheres only[10].

10. Generally, the Sedigraph®, Microtrac™, and screens provided similar size distributions for most of the samples studied.

11. No clear correlation was found between the Sedigraph®, the Microtrac™, the Brinkmann Particle Size Analyzer and Image Analyzer, and conventional microscreens; independent studies have confirmed this finding[9,10].

12. In general, a study of particle size measurement must begin with a definition of "size". In fact, Wills states that "...the exact size of an irregular particle cannot be measured ...because so many different values (of length, breadth, thickness, or diameter) can be determined"[11].

Typically, instruments measure some linear quantity such as volume diameter, perimeter diameter, sieve diameter, Feret's diameter, Martin's diameter, Stokes diameter, projected area diameter, etc., and relate this quantity to "size". Clearly, then, when comparing results from different instruments (which measure different quantities), good agreement can be expected only for "spheres, and rapid divergence of results will occur as we move away from sphericity"[9].

8.0 RECOMMENDATIONS

1. When using the Brinkmann LQF cell, use the shortest possible length of tubing, and minimize horizontal runs.

2. Check the condition of the tubing on the Brinkmann LQF cell before use. If it is old and tacky, replace it.

3. Prior to analysis using the Brinkmann PSA, carefully examine the effect of pump speed on the measured results, and choose an appropriate speed; dense particles will require higher speeds.

4. Use the Sedigraph® to analyze samples that are very fine and homogeneous.

5. When analyzing dense particles on the Sedigraph® and Brinkmann instruments, denser support fluids must be used.

6. Always perform at least three runs per sample and average the results to obtain more accurate measurements.

7. For rapid determination of size distributions in materials having few fine (<2 μm) particles, the Microtrac™ is the instrument of choice.

8. For a more accurate reading on the Microtrac™, use the procedure described by Cameron et al.[2] to calculate the percentage of the sample below 2 μm.

9. While further testwork could be carried out using narrowly-sized standards to better correlate instrumental measurements, such correlation is often unnecessary in mineral processing work. That is, absolute sizing is usually much less important than relative sizing.

   For instance, a rod mill discharge could be measured for, say, four different feed rates, and would result in a set of four distinct distribution curves for each of the instruments compared in this report. From then on, shifts in those curves would reflect changes in ore hardness, or could serve to monitor grinding efficiency, etc. Thus any of the sizing methods considered here could be used, and other considerations such as available sample size, sample composition and density, or speed of analysis take priority in determining the instrument of choice.

APPENDIX A

Summary of Reproducibility of Analyses

Tables 6 and 7 summarize the results of using a given instrument to make several measurements on a given material. At any particular particle size, the resulting "Cumulative % Passing" curves gave a range of values for percent passing at that size. The standard deviation of that range was recorded for each of twelve reference sizes between 2.8 and 176 μm. The highest, lowest, and the average of all twelve standard deviations were recorded in the tables in the "High", "Low", and "Avg." columns, respectively.

Brinkmann PSA dial settings of 3.5, 7, and 10 correspond to circulating pump outputs of 130, 333, and 500 mL/min.

Table 6: Standard Deviations of Percent Passing for Repeat Analyses of the Same Sample

Method	Glass Beads			Sphalerite			Hematite		
	High	Low	Avg.	High	Low	Avg.	High	Low	Avg.
Sedigraph®	2.47	0.71	1.62	1.41	0.71	1.06	4.95	0.0	2.4
Microtrac®	2.48	0.10	0.67	1.67	0.09	0.44	1.77	0.0	1.66
Brinkmann PSA 3.5*	-	-	-	-	-	-	-	-	-
Brinkmann PSA 7*	2.82	0.0	1.18	10.6	0.70	3.5	14.1	0.0	3.4
Brinkmann PSA 10*	-	-	-	-	-	-	-	-	-
Brinkmann Image An.	6.4	0.0	1.76	-	-	-	6.4	0.0	1.8

* circulating pump speed (see p. 27)

Table 7: Standard Deviations of Percent Passing for Repeat Analyses of Fresh Samples

Method	Glass Beads			Sphalerite			Hematite		
	High	Low	Avg.	High	Low	Avg.	High	Low	Avg.
Sedigraph®	4.1	2.1	2.9	7.6	0.9	4.0	8.6	0.6	4.4
Microtrac®	1.6	0.7	1.3	7.5	0.7	4.4	8.4	2.2	3.7
Brinkmann PSA 3.5*	30.4	2.8	25.9	7.1	0.0	1.6	10.4	2.8	8.9
Brinkmann PSA 7*	5.7	1.5	3.4	10.1	0.5	6.8	25.9	0.9	18.7
Brinkmann PSA 10*	-	-	-	-	-	-	42.8	2.9	31.2
Brinkmann Image An.	12.7	2.1	8.0	11.3	0.0	3.6	21.9	1.0	9.2

* circulating pump speed (see p. 27)

APPENDIX B

Summary of Measured Size Distributions

MATERIAL	TABLE	PAGE
Calcite Fines	8	30
Calcite Mids	9	30
Calcite Coarse	10	30
Calcite Heads	11	31
Glass Beads	12	31
Sphalerite	13	32
Hematite	14	32

Table 8: Summary of Results for Calcite Fines

Method	50% Passing	80% Passing	Maximum
Brinkmann Cuvette	7.0 µm	14.2 µm	100 µm
Brinkmann LQF	6.5 "	13.7 "	140 "
Brinkmann IA	14.0 "	19.8 "	40 "
Sedigraph®	4.6 "	7.8 "	40 "
Microtrac™	3.5 "	7.5 "	23 "
Micro Screens	11.5 "	18.6 "	40 "

Table 9: Summary of Results for Calcite Mids

Method	50% Passing	80% Passing	Maximum
Brinkmann Cuvette	25 µm	47 µm	150 µm
Brinkmann LQF	18 "	33 "	150 "
Brinkmann IA	28 "	35 "	70 "
Sedigraph®	16 "	22 "	40 "
Microtrac™	18 "	24 "	45 "
Micro Screens	16 "	23 "	42 "

Table 10: Summary of Results for Calcite Coarse

Method	50% Passing	80% Passing	Maximum
Brinkmann Cuvette	34 µm	47 µm	120 µm
Brinkmann LQF	34 "	53 "	150 "
Brinkmann IA	55 "	70 "	100 "
Sedigraph®	-	-	-
Microtrac™	27 "	53 "	90 "
Micro Screens	32 "	40 "	(80) "

Table 11: Summary of Results for Calcite Heads

Method	50% Passing	80% Passing	Maximum
Brinkmann Cuvette	11 μm	24 μm	100 μm
Brinkmann LQF	13 "	30 "	140 "
Brinkmann IA	40 "	56 "	160 "
Sedigraph®	20 "	27 "	(80)† "
Microtrac™	10 "	22 "	90 "
Micro Screens	20 "	28 "	(70)† "

†Bracketed numbers are extrapolated values.

Table 12: Summary of Results for Glass Beads

Method	50% Passing	80% Passing	Maximum
Brinkmann 3.5*	19 μm	36 μm	90 μm
Brinkmann 7*	17 "	29 "	100 "
Brinkmann 10*	13 "	20 "	80 "
Brinkmann IA	37 "	51 "	150 "
Sedigraph®	26 "	33 "	60 "
Microtrac™	27 "	37 "	90 "
Micro Screens	33 "	41 "	90 "

* circulating pump speed (see p. 27)

Table 13: Summary of Results for Sphalerite

Method	50% Passing	80% Passing	Maximum
Brinkmann 3.5*	5.3 μm	8 μm	50 μm
Brinkmann 7*	7.9 "	18 "	120 "
Brinkmann 10*	6.3 "	12 "	30 "
Brinkmann IA	23 "	48 "	60 "
Sedigraph®	15 "	29 "	60 "
Microtrac™	17 "	35 "	120 "
Micro Screens	19 "	34 "	60 "

* Circulating pump speed (see p. 27)

Table 14: Summary of Results for Hematite

Method	50% Passing	80% Passing	Maximum
Brinkmann 3.5*	20 μm	70 μm	150 μm
Brinkmann 7*	61 "	120 "	250 "
Brinkmann 10*	50 "	100 "	220 "
Brinkmann IA	70 "	120 "	220 "
Sedigraph®	17† "	30† "	130† "
Microtrac™	38 "	65 "	130 "
Micro Screens	45 "	80 "	200 "

† Invalid analysis due to attraction of magnetics to stirring bar.

* Circulating pump speed (see p. 27)

Figure 14: Size Distribution of Calcite - NORMAL Scale

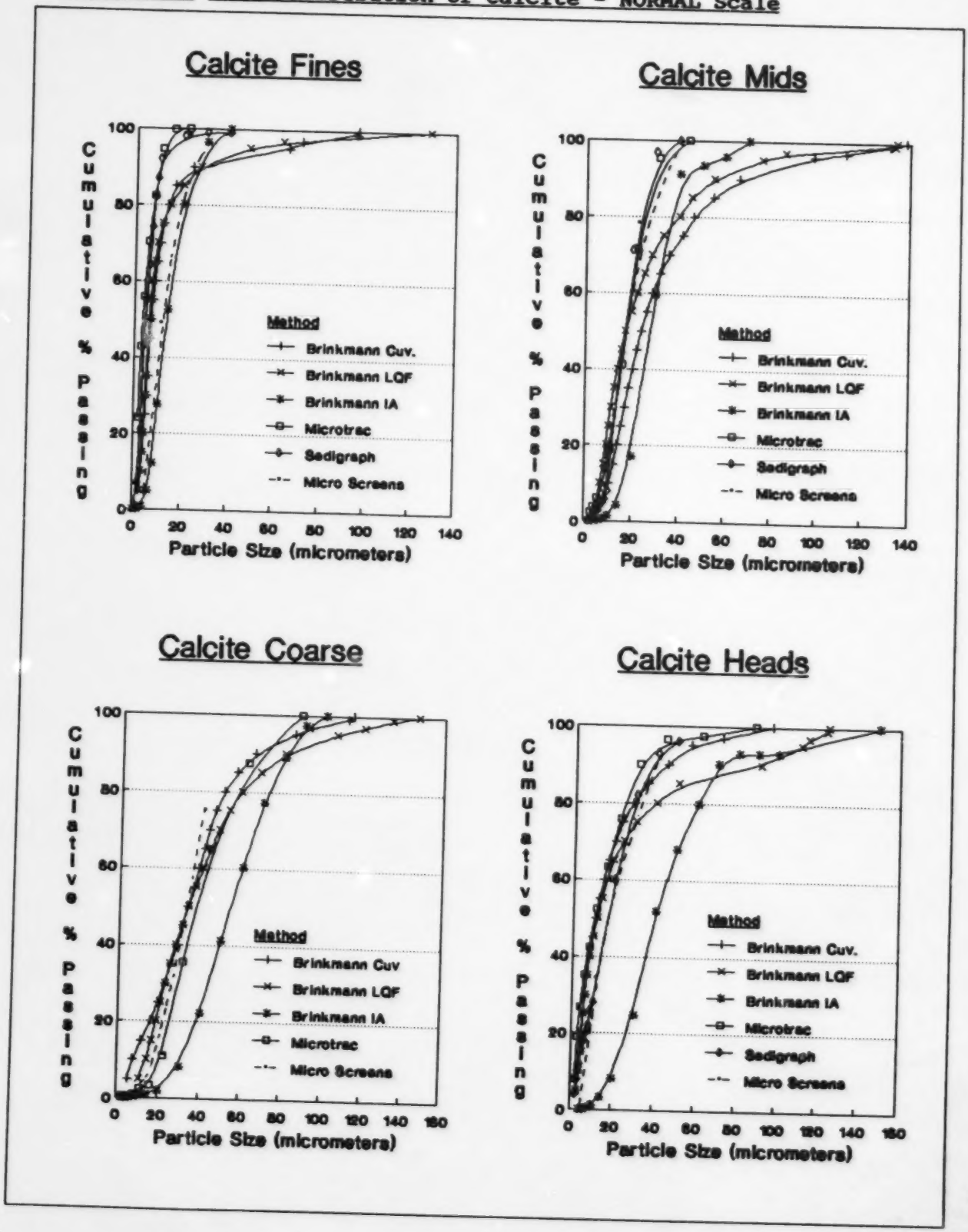
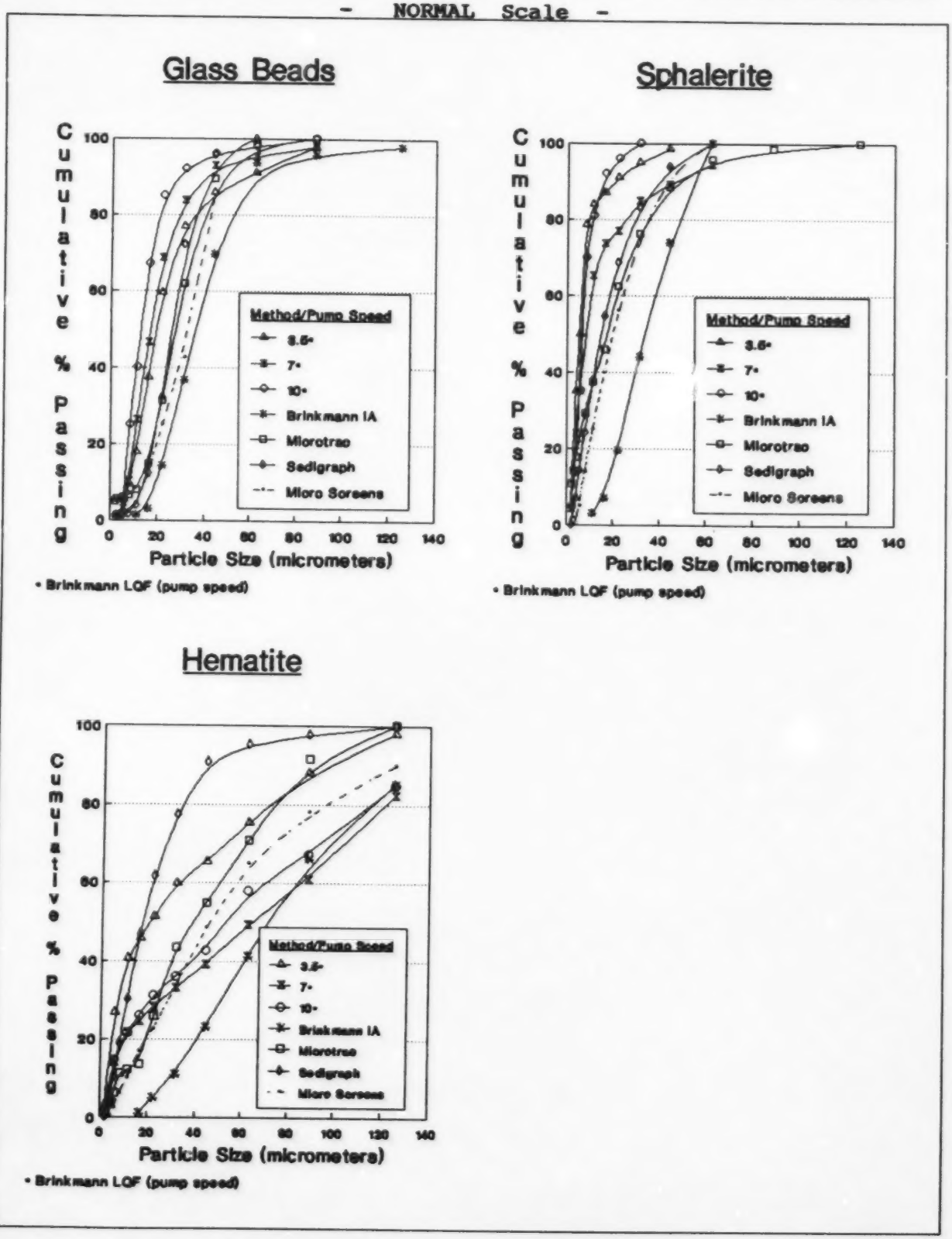


Figure 15: Size Distribution of Glass Beads, Sphalerite, and Hematite

- NORMAL Scale -



APPENDIX C

Comparison of Particles with Equal Volumes but Different Shapes

[Table of "diameters" of Particles of Equal Volume (1 mm^3) but Different Shape][5]

SHAPE	DESCRIPTION	CALCULATED SURFACE AREA (mm^2)	DIAMETER OF A SPHERE (mm)			RATIO: d_M/d_{scr}
			HAVING THE SAME SURFACE AREA AS PARTICLE d_A	SHOWING THE SAME PROJECTED AREA AS PARTICLE (Sizing by Microscope or Image Analysis) d_M	PASSING THE SAME MINIMUM SQUARE APERTURE (Screen Analysis) d_{scr}	
Sphere	-	4.84	1.24 = d	1.24	1.24	1.00
Icosahedron	20 equilateral triangles	5.15	1.28	1.32	1.23	1.07
Dodecahedron	12 pentagons	5.31	1.30	1.35	1.27	1.06
Cube-octahedron	6 octagons and 8 equilateral triangles	5.69	1.35	1.14	-	-
Octahedron	8 equilateral triangles	5.72	1.35	1.36	1.24	1.10
Tetrahedron	4 equilateral triangles	7.20	1.51	1.51	1.44	1.05
Plate(5:5:1)	-	8.19	1.61	1.93	1.45	1.33
Needle(10:1)	right circular cylinder	8.35	1.63	1.80	0.50	3.60*

* Note the large discrepancy in measured values for acicular particles and these two methods.

APPENDIX D

Definitions

Definitions:

Aspect ratio - The ratio of the maximum linear diameter to the minimum linear diameter of a particle, or the ratio of the largest to the smallest of the Feret's diameters measured.

The "d_{75}" value - the size at which 75% of the members of a given population are included in the "percent passing".

Feret's diameter - The mean value of the distance between pairs of parallel tangents to the projected outline of a particle, measured at 0°, 45°, 90°, or 135° to a reference diameter.

Graphical slope - the measured vertical distance between the d_{75} and the d_{50}, divided by the measured horizontal distance between the same points. A large value depicts a narrow distribution.

Martin's diameter - the mean chord length of the projected outline of a particle.

Mathematical slope - the rise over the run between d_{75} and d_{50}, using the values on the axes of a graph. A large value depicts a narrow distribution.

Shape factor - a measure of the sharpness of an object, where a value of 1 corresponds to a circle, and values approaching 0 indicate straight lines (i.e. - having length but no thickness) - equal to $(\text{area}/\text{perimeter}^2) \times 4\pi$.

Stokes diameter - the diameter of a sphere of the same material and having the same sedimentation velocity as a given particle.

References:

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4. "Appendix 2 - Sedimentation Theory" Micromeritics Sedigraph® 5000ET Particle Size Analyzer Operating Manual.

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